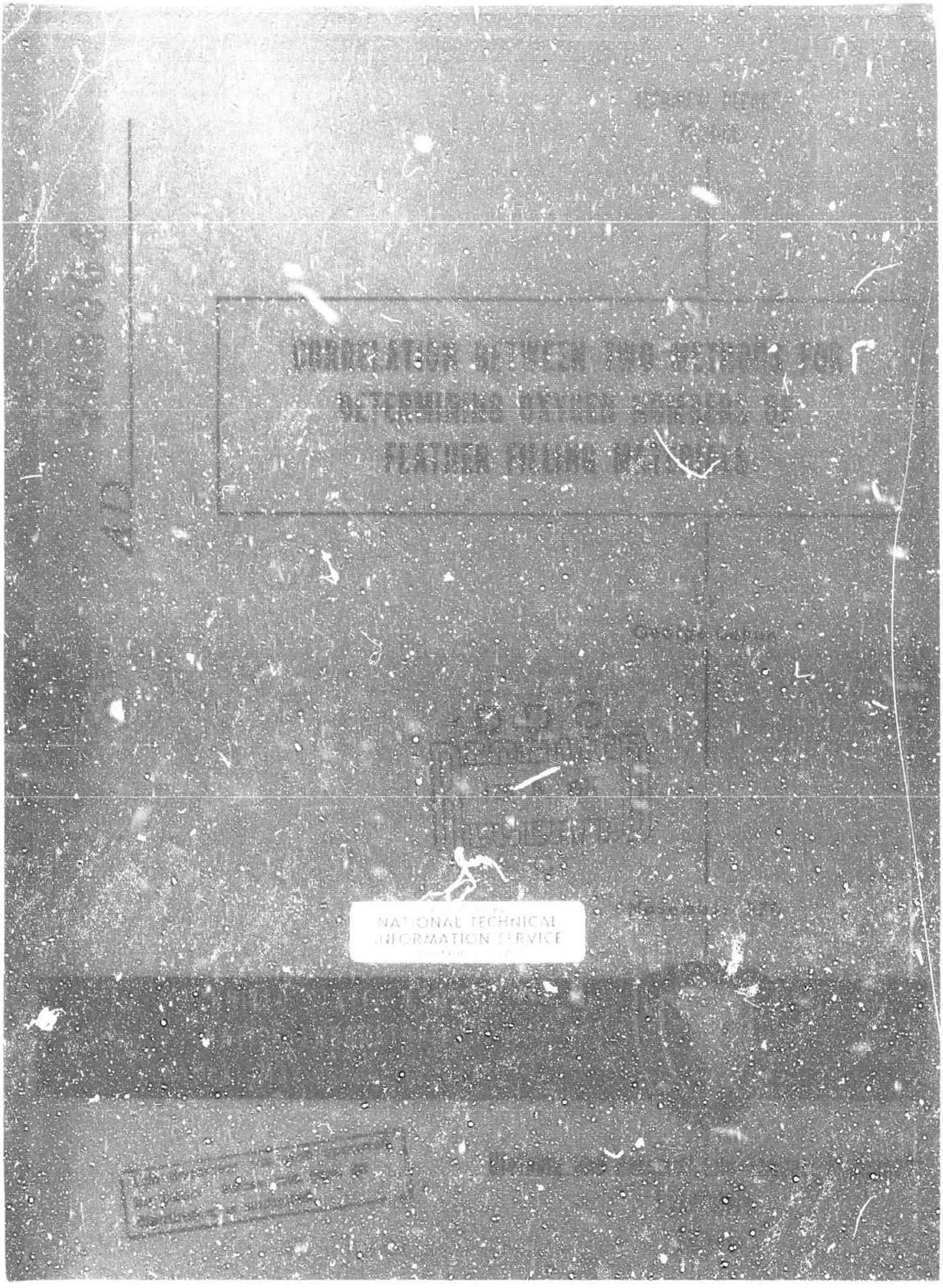

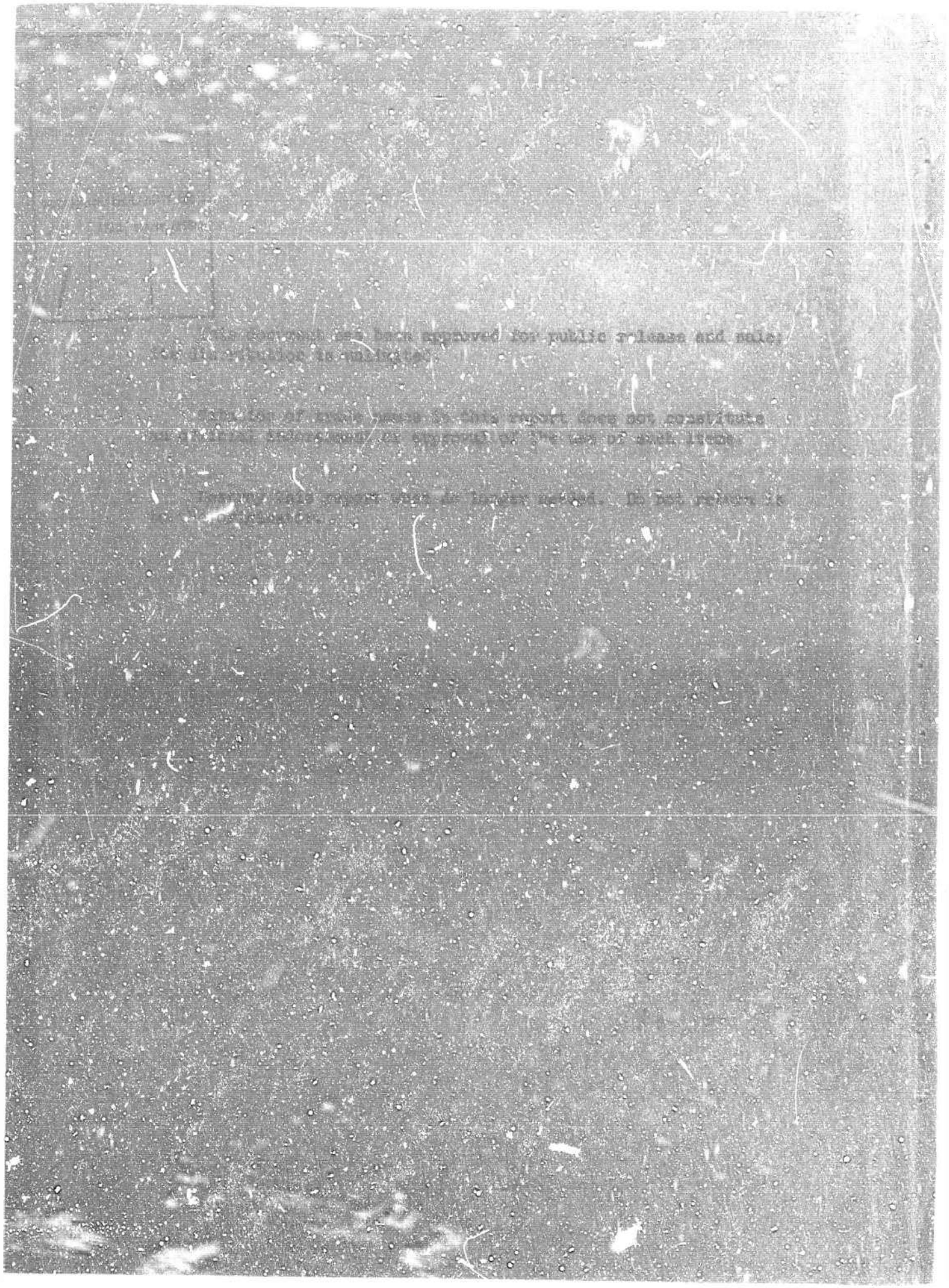

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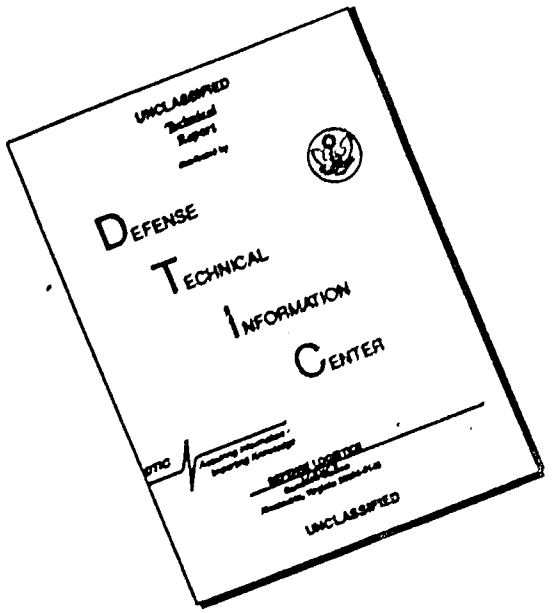

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TECHNICAL REPORT

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CORRELATION BETWEEN TWO METHODS FOR DETERMINING OXYGEN NUMBERS OF FEATHER FILLING MATERIALS

by

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Project Reference:

10342450

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FOREWORD

This report gives the results of a study carried out to determine the correlation between two methods for determining the cleanliness (oxygen number) of feather filling materials. Both methods are widely used by the various state bedding law enforcement agencies and the Federal Government. No systematic attempt had been made previously to establish the equivalency of the resulting data. This study shows that the two methods have a high degree of correlation and the relationship between them can be expressed by a regression equation. This work was carried out by the Textile Engineering Section, Fiber and Fabric Research and Engineering Branch, Textile Research and Engineering Division of the Clothing and Personal Life Support Equipment Laboratory under Code 10342450, Refinement of Specific Test Methods.

Special acknowledgment is made to Dr. S. J. Kennedy, Director, Clothing and Personal Life Support Equipment Laboratories, for his direction of the study and to Mr. Howard C. Winslow, Assistant Chief of Furniture and Bedding Inspection, State of California, whose laboratory made oxygen number determinations by one of the two methods studied.

CONTENTS

LIST OF TABLES

LIST OF FIGURES

ABSTRACT

The correlation between two methods of determining the cleanliness of feather filling materials has been determined. Both methods determine the "oxygen number" by titrating a water extract of the filling materials with a dilute potassium permanganate solution. The methods differ in the manner of preparing the water extract, and determination of the end point. In one method used by the Federal Government, the endpoint is determined visually. In the other method, used by the Laboratory of the Bureau of Furniture and Bedding Inspection, State of California, the endpoint is determined by a colorimeter. It was found that the two methods have a high degree of correlation, with a correlation coefficient of 0.982. The following regression equation shows the relationship between the two procedures where Q is the theoretical oxygen number according to the Government method and C is the actual oxygen number determined by the California method:

$$Q = 0.905C + 1.72$$

All of the actual values of Q were within two standard errors of the estimated theoretical values calculated by this equation.

CORRELATION BETWEEN TWO METHODS FOR DETERMINING OXYGEN NUMBERS OF FEATHER FILLING MATERIALS

Introduction and Objectives

Because of their excellent filling power and insulating qualities, feathers and down have been used for generations in pillows, sleeping bags and clothing. These materials generally are divided into three broad categories: waterfowl feathers, down and landfowl feathers. Waterfowl feathers can be further classified according to their origin as either goose or duck, and landfowl feathers as chicken or turkey. Down, due to its unique structure, is extremely light in weight with high filling power and excellent resilience. Waterfowl feathers differ from landfowl feathers primarily in that they have a pronounced curl which gives them their improved filling power. To improve filling power, landfowl feathers are usually put through a machine which crushes or breaks the shaft of the feathers. In this form they are known as crushed feathers. Down is by far the most expensive and landfowl feathers the least expensive of these materials.

Until the development of the Tan-O-Quil-QM process[1] by the U. S. Army Natick Laboratories, down and feathers, except for washing, had been used in their natural state. Feathers treated by this process have greatly improved filling power, cleanliness and resistance to degradation. The process has been widely adopted by the feather and down industry. All feathers and down procured by the U. S. Army for sleeping bags are treated.

Feathers and down, in their natural state, contain foreign matter consisting of blood, soil, vegetable and fecal matter. Fortunately, these materials are easily removed by a relatively simple procedure of washing with a detergent and an alkaline builder followed by several rinses and a laundry sour. Washing and drying procedures for these materials have been described by Cohen[1] and Rogers, Kaplan and Cohen[2]. The latter authors found that a commercial washing and drying procedure properly carried out, readily removed or destroyed the natural bacteria found on feathers, with the exception of the spore forming bacilli.

To determine if the feathers or down have been properly washed, a method of determining the cleanliness is required. In 1928, N. F. Knight[3] described a method for determining the cleanliness of feathers. It consisted of obtaining an "oxygen number" by determining the amount of potassium permanganate required to cause a pink color to persist in a water extract of the feathers. In 1941 Kane, Pomerantz and Hechtman[4] evaluated various methods. Based upon their work, the State of New York adopted a method for determining the cleanliness of feathers which consisted essentially of titrating a water extract of the feathers with a 0.1 normal potassium permanganate solution. W. R. Goerz[5] in 1946 proposed a similar method which was used by the State of California. This method was later revised by H. C. Winslow so that the endpoint was determined by a colorimeter and recorder rather than visually. In 1949 Brigandi and Natrice[6] investigated various methods of

determining the cleanliness of feathers and concluded that only the oxygen number method gave consistent and reproduceable results. This method is used in Military and Federal Specifications[7] and by the State Bedding Law Officials.[8] The State of California used the revised method developed by Goerz and Winslow.[9] Both methods, as contained in Federal Standard 148a, are given in the Appendix. In the remainder of this report, these will be refered to as methods 4 and 12, respectively, corresponding to the nomenclature in Federal Standard 148a.

Methods 4 and 12 are similar in many respects. In both methods a 10 gram sample of feathers or down is tumbled in a jar with 1 liter of distilled water and the extract is filtered and titrated with 0.1 normal potassium permanganate solution. They differ, however, in the following manner:

a. The revolutions per minute (R. P. M.) of the tumbler jar is 55 R. P. M. for method 4 and 88 R. P. M. for method 12.

b. The total time of tumbling is 60 minutes for method 4 and 15 minutes for method 12.

c. The size of the screen openings through which the extract is filtered, is 74 microns (standard no. 200) for method 4 and 420 microns (standard no. 40) for method 12.

d. In method 4 the end point is determined visually while in method 12 it is determined with a colorimeter.

Most of the State Laboratories use the official method of analysis of the Association of Bedding and Furniture Law Officials[8] This is similar to that of method 4 except that the rate and time of tumbling are followed from method 12. Requirements for oxygen numbers vary among the various states and Federal organizations. In general, however, oxygen numbers greater than 24 indicate the filling material has not been properly washed. Requirements in Military and Federal specifications vary from 6 for Tan-O-Quil-QM treated waterfowl feathers and down to 12 for untreated crushed chicken feathers.

The Laboratory of the Bureau of Furniture and Bedding Inspection, State of California, recognized as outstanding in the field, has conducted studies on feathers and down for the Federal Government and the Association of Bedding and Furniture Law Officials. Since cleanliness of feathers and down, as determined by the oxygen number is a requirement of the Federal Government and of many of the states, it was considered desirable to determine if methods 4 and 12 could be used interchangeably, and if not, what the relationship was between them. This paper gives the results of a study designed to answer these questions.

2 Procedure

Three laboratories participated in this program. Each laboratory made ten determinations of oxygen number on each of the eight types of feathers

and down listed in Table I. Two of these laboratories, coded Q and L, used method 4 and one coded C, used method 12. Q is the laboratory at the U. S. Army Natick Laboratories, L is a commercial laboratory and C is the laboratory of the Bureau of Bedding and Furniture Inspection, State of California. The eight types of material analyzed were selected as being representative of various types of commercial materials and also to give a range of oxygen numbers.

TABLE 1

Types of Filling Material Analyzed

Sample No.	Description
634	Untreated Whole Chicken Feathers
635	Tan-O-Quil-QM Treated Whole Chicken Feathers
636	Untreated Waterfowl Feathers
637	Mixture of 40% Down, 60% Waterfowl Feathers (Untreated)
638	Untreated Down
639	Tan-O-Quil-QM Treated Waterfowl Feathers
640	Untreated Crushed Chicken Feathers
641	Tan-O-Quil-QM Treated Crushed Chicken Feathers

To prepare uniform samples for analysis, one pound of material was placed in a cylindrical wire cage and rotated for one hour at 10 R. P. M. The interior of the cage was fitted with baffles which moved the material from side to side in addition to the forward movement in the direction of rotation. The material was then piled on a table in the form of a cone and divided into three vertical sections or slices, one of which was sent to each laboratory for analysis.

3. Results and Discussion

The oxygen numbers determined by each laboratory and the averages for the ten determinations made on each sample, are tabulated in Table II. In the remainder of this report, unless otherwise stated, the values used for oxygen numbers are the averages of the set of ten determinations made on each sample by a laboratory.

TABLE II
Oxygen Numbers

Sample No.	Laboratory	1	2	3	4	5	6	7	8	9	10	Avg.
634	C	43.2	48.0	57.2	51.2	53.0	43.2	54.0	47.2	56.8	43.6	49.7
	Q	52.0	54.0	52.0	52.0	44.0	48.0	64.0	50.0	44.0	44.0	50.4
	L	48.0	48.0	48.0	52.0	44.0	48.0	48.0	48.0	48.0	44.0	47.6
635	C	5.0	4.8	5.6	5.6	5.2	5.2	6.0	6.0	4.4	6.0	5.4
	Q	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
	L	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
636	C	6.4	5.6	5.2	5.6	6.0	6.0	6.0	6.0	4.8	4.8	5.6
	Q	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0
	L	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0
637	C	11.0	11.9	11.2	11.4	16.0	12.0	9.2	11.4	10.4	11.2	11.6
	Q	15.3	20.8	24.0	16.0	14.0	17.0	16.0	14.0	25.0	22.0	18.4
	L	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0
633	C	32.0	36.4	34.0	37.6	36.0	38.4	36.0	38.4	28.0	28.8	34.6
	Q	32.0	31.0	34.0	31.0	32.0	31.0	36.0	35.0	34.0	36.0	33.2
	L	28.0	32.0	32.0	32.0	32.0	32.0	32.0	32.0	28.0	28.0	30.8
639	C	6.8	4.4	4.4	4.4	6.4	4.8	6.0	3.6	6.0	4.8	5.2
	Q	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
	L	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
640	C	42.4	46.8	58.8	57.2	54.0	54.4	54.4	52.0	61.6	49.2	53.1
	Q	44.0	40.0	54.0	40.0	48.0	46.0	47.0	44.0	44.0	51.0	45.4
	L	52.0	52.0	52.0	60.0	56.0	56.0	60.0	60.0	56.0	60.0	56.4
641	C	10.0	5.2	10.8	12.0	10.4	10.0	12.8	5.2	11.2	10.8	9.8
	Q	8.0	8.0	8.0	8.0	8.0	10.0	9.0	8.0	8.0	8.0	8.3
	L	11.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0	16.0

The data in Table II shows that all of the individual determinations of oxygen numbers made by laboratory L were multiples of 4. Also that for five out of the ten sets of determinations made, the 10 indivudal readings were identical. Since one drop of 0.1 permanganate is approximately .05 ml or an oxygen number of 4, it is indicated that in these cases the oxygen number was determined counting the number of drops of permanganate added and multiplying by 4. Method 4 requires that the amount of permanganate used be determined by readings on a burette calibrated to .02 ml. Nevertheless, a high degree of correlation was obtained between laboratories L and C or Q.

The regression equation between laboratories Q and C calculated by the method of least squares [11] was $Q=0.905C + 1.72$. Using this equation, values for Q (Table IV) were calculated. The sum of the differences between laboratories using actual and calculated values of oxygen numbers (Table IV), was found to be about the same (-4.3 vs -4.6) indicating that the overall differences in either case were about equal. The standard error of estimate [12] was 3.4. Theoretically for a normal distribution, 95 percent of the values should fall within two standard errors of estimate of the calculated values. Actually (Table IV) all of the values were well within this limit. This is shown graphically in Figure 4.

The relation between the oxygen numbers, as determined by each laboratory is shown graphically in Figures 1, 2 and 3. There appears to be a high degree of correlation between the various laboratories. This was confirmed by the correlation coefficients given in Table III. These were calculated using the procedure of Maroney [10].

TABLE III

Correlation Coefficients - Between Laboratories

Laboratories	Correlation Coefficient
Q vs L	.938
C vs L	.960
C vs Q	.982

4. Conclusions

There is a high degree of correlation between methods 4 and 12 in Federal Standard 148a, for determining oxygen numbers. The following regression equation appears to fit the data as all of the actual values are within two standard errors of estimate of the calculated value.

$$Q = 0.905C + 1.72$$

C is the oxygen number determined by method 12 and Q is the value according to method 4.

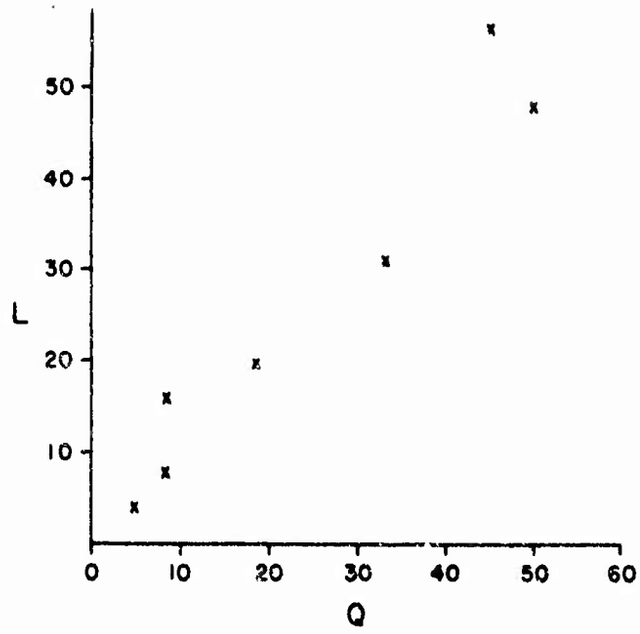

Figure 1. Oxygen Numbers Q vs. L

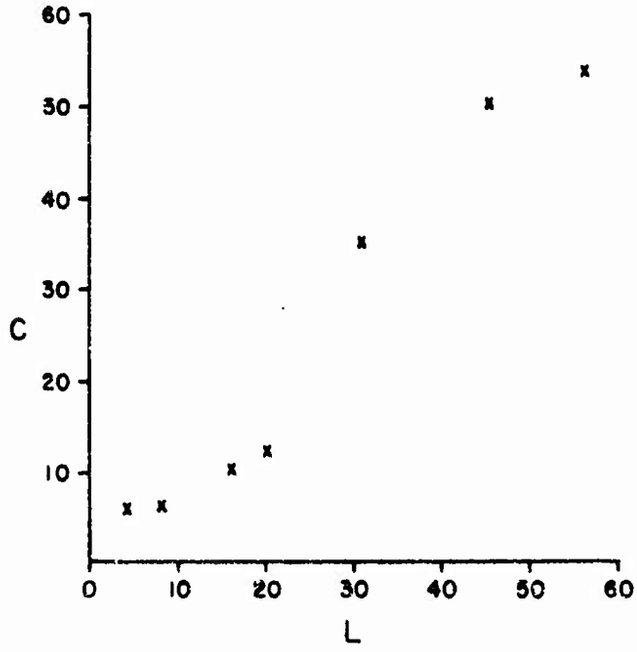

Figure 2. Oxygen Numbers L vs. C

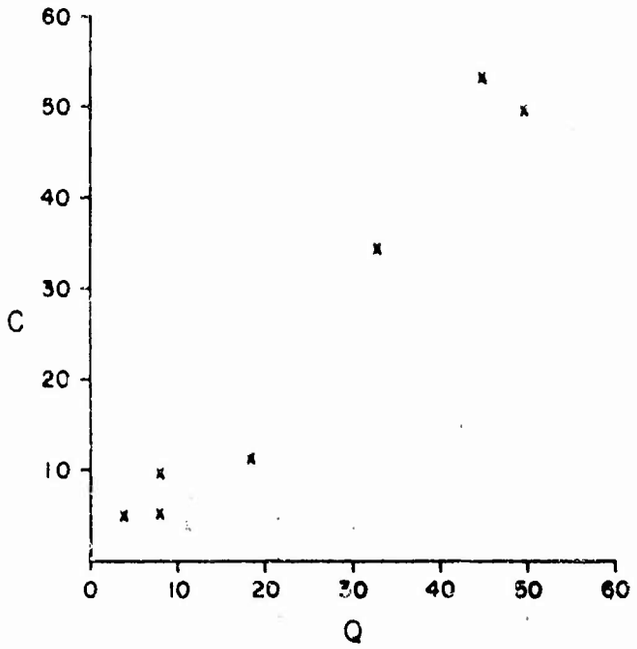

Figure 3. Oxygen Numbers Q vs. C

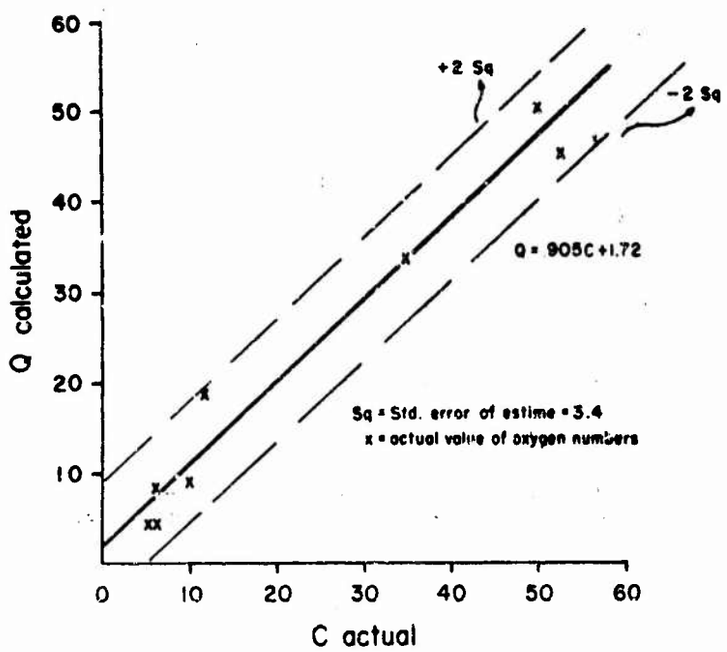

Figure 4. Oxygen Numbers C Actual vs. Q Calculated

TABLE IV

Comparison of Actual and Calculated* Values of Oxygen Numbers

	Oxygen Numbers								Total
Sample No.	634	635	636	637	638	639	640	641	
Laboratory -									
C actual	49.7	5.4	5.6	11.6	34.6	5.2	53.1	9.8	
Q actual	50.4	4.0	8.0	18.4	33.2	4.0	45.4	8.3	
Q calculated*	46.7	6.6	6.8	12.2	33.0	4.9	49.7	10.5	
Q act - C act	+0.7	-1.4	+2.4	+6.8	-1.4	-1.2	-8.7	-1.5	-4.3
Q cal*- C act	-3.0	+1.2	+1.2	+0.6	-1.6	-0.3	-3.4	+0.7	-4.6
Q cal*- Q act	-3.7	+2.6	-1.2	-6.2	-0.2	+0.9	+4.3	+2.2	-1.3

* Calculated from Q = .905C + 1.72

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Appendix

DETERMINATION OF OXYGEN NUMBER (TITRATION METHOD)

1. SCOPE

1.1 This method is intended for determining the oxygen number of feathers, feather products, down and mixtures thereof by means of a titration process.

2. TEST SPECIMEN

2.1 The specimen shall consist of 10.0 ± 0.1 grams of material prepared as specified in 5.1.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens shall be tested from each sample unit.

4. APPARATUS AND REAGENTS

4.1 Apparatus

4.1.1 Tumbler jar. The tumbler jar and apparatus shall be as specified in method 5500 of Federal Specification CCC-T-191, except that the jar shall be all glass or stainless steel.

4.1.2 74 micron (standard No. 200) sieve

4.1.3 Analytical balance

4.1.4 Micro-burettes (2) (divided into 0.02-ml divisions).

4.1.5 Porcelain casserole

4.1.6 Stopwatch or other suitable equivalent, and timer

4.1.7 Beaker, 2,000 milliliters

4.2 Reagents

4.2.1 Distilled water

4.2.2 6N sulfuric acid

4.2.3 Potassium permanganate

5. PROCEDURE

5.1 Preparation of specimen. Approximately 28 grams of the material shall be exposed in the unpacked state in a container composed of solid bottom with screened sides and top until in standard condition. The material shall be mixed with a rod over the exposure period to insure complete relaxation and conditioning. The test specimen consisting of 10.0 ± 0.1 grams shall be taken from the conditioned material.

5.2 The specimen shall be placed in a tumbler jar with 1 liter of distilled water, sealed and tumbled at room temperature for 60-65 minutes. The resulting suspension shall be filtered through a 74 micron (Standard No. 200 sieve) into a beaker. Do not squeeze excess water from stock into beaker.

5.3 A 100-ml. aliquot of the above filtrate shall be transferred to a porcelain casserole, neutralized and made acid with the addition of 1 to 2 milliliters excess of 6N sulfuric acid. The solution shall be titrated with standard 0.1N potassium permanganate, by means of a burette divided into 0.02-ml. divisions, adding approximately 0.02 ml. at a time until a pink color persists for 60 seconds. This small amount is not sufficient to make a full drop and shall be collected on a glass stirring rod and then added to the solution. Calculate oxygen number to the number of grams of oxygen per 100,000 grams of the sample as follows: The number of milliliters at 0.1N K MnO_4 determined shall be multiplied by a constant (80). The product from the multiplication shall be considered "the initial oxygen number of the feather material" and in calculation of results is indicated as "A".

5.3.1 Blank determination distilled water. A blank determination shall be made on distilled water to determine the oxygen number of the water. This shall be done by following the exact procedure stated above excluding the feather material. The value determined shall be considered "the blank determination of the distilled water" and in the calculations of results is indicated as "B".

6. CALCULATION OF RESULTS

6.1 The true oxygen number of the feather material shall be calculated from the follwoing formula:

True oxygen number of feather material =A-B
Where:
A=Initial oxygen number of the feather material
B=Blank determination of the distilled water

7. REPORT

7.1 The true oxygen number of the sample unit shall be the average of the true values obtained from the specimens tested and shall be reported to the nearest whole number.

Appendix

DETERMINATION OF OXYGEN NUMBER COLORIMETER METHOD

1. SCOPE

1.1 This method is intended for determining the oxygen number of filling materials. In the interest of standardization of testing requirements, it is recommended that this method not be used in procurement documents.

2. TEST SPECIMEN

2.1 The specimen shall consist of 10.0 ± 0.1 grams of material prepared as specified in 5.1.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens shall be tested from each sample unit.

4. APPARATUS AND REAGENTS AND METHODS CITED

4.1 Apparatus

4.1.1 Tumble jar. The tumble jar shall be as specified in method 5500 of Federal Specification CCC-T-191 except that the speed of the bar shall be adjusted to 88 ± 3 revolutions per minute. An equivalent agitating device may be used, but care should be exercised in its selection as the agitation of the material influences the results of the test.

4.1.2 420 micron (Standard No. 40) sieve.

4.1.3 Analytical balance

4.1.4 Micro-burettes (2).

4.1.5 Colorimeter. Photovolt Colorimeter Model 401-T with filter #530 or equivalent.

4.1.6 Recorder. Varian Model G11A Strip Chart Recorder set to 25 mv span with chart speed of 2 inches per minute and chart paper type 5A or equivalent.

4.1.7 Stopwatch or suitable equivalent

4.1.8 Beaker. 2,000 ml.

4.1.9 Graduates. 1,000 ml., 100 ml.

4.2 Reagents

4.2.1 Distilled water

4.2.2 6N sulfuric acid

4.2.3 0.1N potassium permanganate

5. PROCEDURE

5.1 Preparation of specimen. Approximately 28 grams of the material shall be exposed in the unpacked state in a container with a solid bottom and screened sides and top until in standard condition. The test specimen consisting of 10.0 ± 0.1 grams shall be taken from the conditioned material.

5.2 The specimen shall be placed in a tumble jar with one liter of distilled water and tumbled at room temperature for 15 minutes. The resulting suspension shall be filtered through a 420 micron (Standard No. 40) sieve into a 2,000-ml. beaker. The stock will be captured by the screen sieve and the wash liquor will pass through into the beaker. Do not squeeze excess water from stock into bea

5.3 Remove a 200-ml. aliquot of the filtrate from the 2,000-ml. beaker and pl a colorimeter cell. Add 2 ml. of 6N sulfuric acid. (Note: If colorimeter and recorder used are of the type that requires a warm up period, the apparatus shall be started and allowed to operate for a period of 5 minutes before use). Turn on stirrer on colorimeter and switch recorder chart switch from stand-by to low.

5.4 Adjust colorimeter by fine and coarse adjusting controls until the meter reading on the colorimeter is 90 on the percent transmission scale. Indicator pen on the recorder must agree with percent transmission meter reading. If transmission reading and recording line reading are not the same, adjust recorder until recorder is in balance with transmission reading on colorimeter.

5.5 If wash liquor being tested is too turbid to adjust colorimeter to 90 percent transmission, choose a lower percent transmission to which the colorimeter will adjust. Again, colorimeter and recorder must agree.

5.6 When recorder and colorimeter are in adjustment, by means of a 5-ml. burette divided into 0.02-ml. divisions, add 3 drops of 0.1 N potassium permanganate per minute (utilizing a stopwatch) until the recorder chart paper shows a deviation of not less than 2 lines (numbers) below the original setting, i.e., if original setting was 90, a reading of not more than 88 must be recording at the end of a minute interval before test is stopped. The number of milliliters of potassium permanganate used to indicate a deviation of not more than two lines or numbers within the specified setting shall be multiplied by a constant (40) to determine the oxygen number of the feather material. The resulting product shall be considered "the initial oxygen number of the feather material" and in calculation of results is indicated as "A". The milliliters of 0.1N potassium permanganate used is equal to original reading of 5-ml. burette containing the 0.1 potassium permanganate minus the final reading.

5.7 Blank Determination Distilled Water

A blank determination shall be made on the distilled water. This shall be determined as specified in 5.6, excluding the feather material and agitating period. The value determined shall be considered "the blank determination of the distilled water" and in calculation of results is indicated as "B".

6. CALCULATION OF RESULTS

6.1 The true oxygen number of the feather material shall be calculated from the following formula:

True oxygen number of feather material=A-B
Where: A=Initial oxygen number of the feather material
B=Blank determination of the distilled water

7. REPORT

7.1 The true oxygen number of the sample unit shall be the average of the true values obtained from the specimens tested and shall be reported to the nearest whole number.

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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
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13. ABSTRACT

The correlation between two methods of determining the cleanliness of feather filling materials has been determined. Both methods determine the "oxygen number" by titrating a water extract of the filling materials with a dilute potassium permanganate solution. The methods differ in the manner of preparing the water extract, and determination of the end point. In one method used by the Federal Government, the endpoint is determined visually. In the other method, used by the Laboratory of the Bureau of Furniture and Bedding Inspection, State of California, the endpoint is determined by a colorimeter. It was found that the two methods have a high degree of correlation, with a correlation coefficient of 0.982. The following regression equation shows the relationship between the two procedures where Q is the theoretical oxygen number according to the Government method and C is the actual oxygen number determined by the California method:

$$Q = 0.905C + 1.72$$

All of the actual values of Q were within two standard errors of the estimated theoretical values calculated by this equation.

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Correlation	8					
Tests	8,9					
Cleaning	9					
Feathers	9					
Oxygen Numbers	10					